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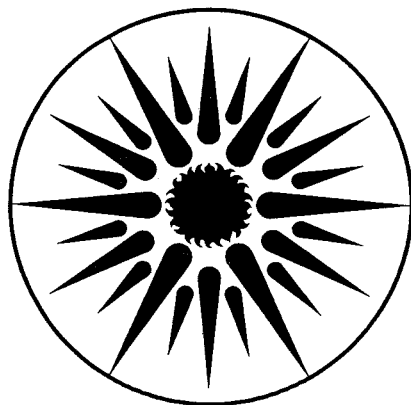
ENERGY & ENVIRONMENT DIVISION

Submitted to American Industrial Hygiene Association Journal

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February 1995



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An Improved Impregnated-filter Method for Measuring Low-level Concentrations of Toluene Diisocyanates in Air

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The project was funded with Federal funds from the U.S. Consumer Product Safety Commission and the Office of Atmospheric and Indoor Air Programs of the U.S. Environmental Protection Agency under interagency agreements, CPSC-IAG-92-1134 and CPSC-IAG-92-1120, as well as the Assistant Secretary for Conservation and Renewable Energy, Office of Building Technologies, Building Systems and Materials Division of the U.S. Department of Energy under Contract DE-AC03-76SF00098.

ABSTRACT

An improved method was developed and validated for measuring low-level concentrations of toluene diisocyanates (TDIs) in air. The method is based on OSHA Method 42 for industrial applications. Airborne TDIs were trapped on a 25-mm glass-fiber filter impregnated with 50 μg of 1-(2-pyridyl)piperazine. A filter holder for was constructed to minimize contamination and losses of the analytes. The derivatized TDIs were extracted by immersion of the filter in a small volume of solvent. The analysis was performed with a high performance liquid chromatograph equipped with a fluorescence detector and a C₁₈ base-deactivated silica column.

The modified method has a lower limit of quantitation of 0.02 ppb in 15 L of air for both 2,4-toluene diisocyanate (2,4-TDI) and 2,6-toluene diisocyanate (2,6-TDI), which is about a fifteen-fold enhancement over Method 42. The recovery efficiencies and 95% confidence intervals for vapor-spiked filters were 77 ± 6 percent for 2,4-TDI and 69 ± 10 percent for 2,6-TDI. The precision of replicate analyses was ten percent or better. The method was used to screen flexible polyurethane foam for emissions of unreacted TDIs.

INTRODUCTION

Toluene diisocyanates (TDIs) are primarily used as ingredients for manufacturing flexible polyurethane foams (1). The largest markets for flexible foams are furniture, automotive seating, carpet cushions and bedding. TDIs are used in smaller amounts for manufacturing urethane coatings, elastomers, adhesives and sealants. TDI vapors can be generated during the manufacturing, processing and handling of polyurethane products (2). Industrial exposures to TDI vapors are controlled at very low levels because isocyanates are potent respiratory irritants and sensitizers that can cause a variety of respiratory diseases, including isocyanate-induced asthma (3).

One widely recognized and sensitive method for measuring individual isomers of TDI is U.S. Occupational Safety and Health Administration (OSHA) Method 42 (4). Samples are collected on glass-fiber filters impregnated with the derivatizing agent 1-(2-pyridyl)piperazine (1-2PP) and are analyzed by high performance liquid chromatography (HPLC) using a fluorescence detector. The estimated lower limits of quantitation for Method 42 are 2.5 and 2.3 $\mu\text{g m}^{-3}$ (0.36 and 0.32 ppb) in a 15-L air sample for 2,4-TDI and 2,6-TDI, respectively. The documentation for the method shows that recoveries were poor when the vapor-spiked amount of analyte was less than approximately 30 ng, which is equivalent to 0.28 ppb for a 15-L sample (4). In addition, severe tailing of unreacted 1-2PP in the HPLC chromatogram can mask the fluorescence from derivatized TDIs until the corresponding filter-collected masses reach 80 ng, which is equivalent to 0.75 ppb for a 15-L air sample (5). Therefore, it was necessary to improve the method in order to measure low-level concentrations of TDIs in air using small-volume samples.

This paper describes modifications to OSHA Method 42 that produced about a fifteen-fold improvement in the lower limit of quantitation. The amount of 1-2PP on the filter was reduced, sample losses and sources of contamination were minimized, and chromatographic performance was significantly improved. Larger sample volumes and tedious steps to concentrate the sample extract or to fractionate the extract to remove unreacted 1-2PP were avoided. An application is reported in which the modified method was used to screen a polyurethane foam carpet cushion for emissions of unreacted TDIs.

EXPERIMENTAL MATERIALS AND METHODS

Reagents

The isocyanates were 2,4-toluene diisocyanate (98%, Carbolabs Inc.) and 2,6-toluene diisocyanate (97%, Aldrich Chemical Co., Inc.). The organic solvents were chromatographic grade (B&J Brand, Baxter Healthcare Corp.). 1-(2-pyridyl)piperazine (98%), ammonium acetate (99.99%) and dichlorodimethylsilane (99%) were obtained from Aldrich Chemical Co., Inc.

The urea derivatives of the TDIs (TDIUs) were prepared by scaling down the procedures in Method 42 (4). The purity of the TDIUs was assessed by HPLC using a diode-array UV absorbance detector. The analysis of each derivative produced a single chromatographic peak. The absorbance spectrum was scanned at the up slope, apex, and down slope of the peak. The spectra at these locations matched, confirming the purity of each TDIU.

Preparation of Impregnated Filters

Samples were collected on 25-mm diameter borosilicate glass-fiber filters (Part No. 61630, Gelman Sciences) impregnated with 1-2PP. Filters were prepared by dispersing 250 μL of a solution of 0.2 mg mL^{-1} 1-2PP in acetonitrile or dichloromethane over the face of each filter with a pipette yielding 50 μg of 1-2PP per filter, a factor of two reduction in mass compared to Method 42. The filter surface wet evenly. Since the stoichiometric ratio of 1-2PP and TDI is 2:1, the maximum collection capacity of the filter is 26.7 μg of TDI. In practice, 50 percent or more of the derivatizing reagent on the filter should remain unreacted. Therefore, the upper concentration limit is about 125 ppb for a 15-L air sample. Treated filters were dried under a stream of nitrogen and briefly heated in an oven at 65° C. The filters were wrapped in aluminum foil and stored at room temperature.

Sample Holder and Sampling

Impregnated filters were held in a device that was constructed to minimize contamination and losses of the analytes. The base of the holder was a 25.4-mm stainless-steel compression fitting (Part No. SS-1610-6-8, Swagelok Co.). The filter was supported face up in the compression fitting on a 25-mm stainless-steel screen (Part No. 79791, Gelman Sciences). For area sampling, the inlet of the holder was a 40-mm length of 25.4-mm O.D. glass tubing ground flat at the outlet. For this study in which an in-line filter holder was used for sampling, the glass tubing was necked down at the inlet to 6.4-mm O.D. and had a total length of 85 mm. The glass tubing was silanized with dichlorodimethylsilane. It was held in the base with Teflon ferrules (Part Nos. T-1613-1 and T-1614-1, Swagelok Co.). For assembly, a 25-mm O.D. Buna O-ring (size 020) was placed on top of the filter on the screen. The glass tubing was pushed down tightly onto the O-ring while hand tightening the compression fitting. This O-ring was discarded after each use.

Sampling flow rates through a filter of 0.02 - 1.0 L min^{-1} were established with a vacuum pump and an electronic mass-flow controller located downstream of the holder.

Analysis

Analyses were performed using an HPLC system (Model 1090M, Hewlett-Packard Co.) equipped with a binary solvent system, a temperature-controlled column compartment, a diode-array UV detector, and a programmable fluorescence detector with a Xenon lamp (Model 1046A, Hewlett-Packard Co.). The manual injector was equipped with a Tefzel rotor seal and a 5- μL sample loop. Separations were achieved with a C_{18} base-deactivated silica (BDS) microbore column (2 x 150 mm) protected with a similar guard column (Part Nos. 155-46-2-CPG & 842-46-P, Keystone Scientific, Inc.). Two standard C_{18} silica microbore columns (Part No. WAT023655, Waters Corp. & Part No. 202TP5215, The Separation Group) were also used. The mobile phase consisted of a mixture of 35 percent acetonitrile and 65 percent 0.05 M ammonium acetate (adjusted to pH 6.2 with glacial acetic acid). The isocratic mobile phase flow rate was 0.2 mL min^{-1} at a column temperature of 30° C. The optimum fluorescence wavelengths were 240 nm for excitation and 370 nm for emission. The HPLC system was periodically rinsed with water to remove any residual buffer.

Stock standard solutions were prepared by dissolving weighed amounts of the TDIUs in acetonitrile ([free TDI] = 0.3479 x [TDIU]). These were stored in the dark at -10° C. Fresh working solutions were prepared by diluting the stock solutions in HPLC mobile phase.

An impregnated filter was extracted by immersing it for 15 min in 2 mL of mobile phase contained in a 20-mL screw-top scintillation vial. The vial was then gently shaken by hand. An aliquot of the extract was withdrawn with a 3-mL plastic syringe and passed through a syringe filter (Acrodisc LC 13 PVDF, Gelman Sciences) into a 1-mL vial for analysis. The glass vials, syringe and filter were discarded after each use.

Vapor generation

The vapor generation system used for the performance evaluation consisted of two diffusion vials each containing several milliliters of either 2,4-TDI or 2,6-TDI. The necks on the diffusion vials had a 5-mm I.D. and were 50-mm long. Each vial was contained in a glass cell with a Teflon-lined screw top (Part No. 19X-415-000, VICI Metronics). The cells were connected at their outlets with a silanized glass cross fabricated from 6.4-mm O.D. tubing. These connections were made with bored-through Teflon fittings. The cells were immersed in a constant temperature water bath at 30.0 ± 0.2° C. Dry nitrogen at 50 ± 1 cm³ min⁻¹ was constantly swept through each cell. The diffusion rates were gravimetrically calibrated by weighing the vials periodically over about two months.

Impregnated filters were spiked by attaching the sample holder to one of the free branches of the connecting cross. The other free branch was vented. The desired flow rate of 20 cm³ min⁻¹ was established in the sampling line. Sampling was initiated by attaching this line to the outlet of the holder with a quick-connect fitting. Sampling was terminated by disconnecting the fitting. The masses of TDIs spiked onto the filters were controlled by varying the sampling times which typically ranged from 1 - 5 minutes.

RESULTS AND DISCUSSION

Optimization of HPLC Analysis

The two standard C₁₈ silica columns were first tested for the separation of 1-2PP and TDIUs. In each case, significant peak tailing was observed even with a high ionic strength mobile phase. Presumably this was due to the irreversible adsorption of the basic 1-2PP and TDIUs onto the active sites of the bonded stationary phase. The 1-2PP signal could not be discriminated because the fluorescence spectra for both 1-2PP and TDIUs are similar. Therefore, quantitation of the TDIU signals on the large tailing 1-2PP signal was difficult, particularly at low TDIU mass loadings. Schemes were devised to remove excess 1-2PP from the sample extracts; however, a much simpler solution was to use the C₁₈ base-deactivated silica column to improve chromatographic separation. The active sites on the bonded stationary phase of this column are end-capped by the manufacturer with a proprietary pre-treatment. This results in TDIU signals that are symmetrical and well separated from a moderately tailing 1-2PP signal (Figure 1). The two-fold reduction in the mass loading of 1-2PP on the filter was another important factor in achieving good chromatographic separation.

The fluorescence detector was calibrated over a range of 7-600 picograms (pg) as free TDI per 5-μL injection. The peak-height responses for both TDIUs over this mass range had linear regression coefficients of 0.999 and near zero intercepts.

The standard deviation for quadruple analyses of 2 pg of TDI was equivalent to about 0.4 pg. The analytical response for a blank treated filter was equivalent to about 1 pg. Therefore, the limit of quantitation defined as the blank signal plus ten times the standard deviation of the analytical signal is 5 pg of TDI. Since the filter extraction volume was 2 mL and the injection volume was 5 μL, the 5-pg value translates to a sample mass of 2 ng of TDI per sample. This gives a lower limit of quantitation for a 15-L air sample of 0.13 μg m⁻³ or 0.02 ppb, which is a fifteen-fold enhancement over the quantitation limit for Method 42 (4). Even lower limits can be obtained by using larger volume samples.

Minimization of Sampling Losses and Contamination

Method 42 uses 37-mm polystyrene holders. These holders were not evaluated in this study. However, TDI is readily lost to surfaces, and it is possible that there are significant sorption losses on polystyrene. This may explain the large negative intercepts in the curves demonstrating the lower limits of detection for Method 42 (4). To minimize such losses, the filter holder for this study was constructed so that the sampled air only comes in contact with the silanized glass inlet and a small exposed area of the Buna O-ring. Even so, a small amount of TDI was lost to the O-ring. To avoid possible cross contamination during sampling, a new O-ring was used for each sample. Other O-ring materials, such as Viton or Teflon, were not evaluated.

TDIUs also stick to glass surfaces as cross contamination was frequently found to be a problem during sample preparation and analysis. Contamination during sample preparation was eliminated by employing disposable glass vials, syringes, and filters which were only used once. The replacement of the HPLC standard injector rotor seal with the Tefzel seal resulted in fewer contamination problems during analysis.

Vapor Generation

Five periodic weighings of the TDI diffusion sources were made over a 53-day period. The diffusion rates for this period were determined by calculating the linear regressions for plots of emitted mass versus elapsed time. The regression coefficients for 2,4-TDI and 2,6-TDI were 0.999 and 0.994, respectively. The diffusion rates and their 95% confidence intervals (CIs) for 2,4-TDI and 2,6-TDI were 51.3 ± 2.7 and 49.6 ± 7.5 , respectively. Thus, there was higher uncertainty in the diffusion rate of the 2,6-TDI isomer.

The generation of known low concentrations of TDI vapors was difficult due to the small changes in the masses of the diffusion sources and the potential for losses in the system. We hypothesize that such difficulties may have been a major factor contributing to the indicated incomplete recoveries of the TDIs that are discussed below. Sources of potential errors that could result in apparent low recoveries include: 1) sorption loss of TDIs to components of the generation system; 2) hydrolysis of the TDIs to amines in the presence of any moisture in the system; 3) temporal instability of the sources; and 4) inaccuracies in the measurements of the flow rates for dilution and sampling. Further effort is needed to validate the performance of the vapor generation system using diffusion sources.

Performance Evaluation

Impregnated filters were first spiked with an aliquot of a solution of both TDIUs in acetonitrile. The spiked masses were 160 ng of each TDIU. The filters were dried under nitrogen and extracted using different methods. The simplest effective procedure was to immerse the filter in 2 mL of the HPLC mobile phase contained in a 20-mL vial. The extraction efficiency for both TDIUs increased with immersion time and reached nearly 100 percent in 15 minutes. This defined the minimum extraction time for the method. In addition, the quantitative recoveries demonstrated that losses of TDIUs during sample preparation were minimal.

All subsequent performance assessments were conducted using the diffusion sources to spike impregnated filters with TDI vapors. To determine if there were breakthrough losses, a set of filters were vapor spiked with 51 ng of each TDI isomer and then room air at 1.0 L min^{-1} was passed through two or more filters for 0, 5, 10, 15, and 30 minutes. The average recoveries and 95% CIs were 64 ± 3 percent for 2,4-TDI and 62 ± 2 percent for 2,6-TDI. There was no significant loss of either TDI with increasing air volume as would be expected if there were breakthrough losses. Nevertheless, the possibility of breakthrough losses was further investigated by placing two impregnated filters in series in the filter holder. A high loading of 220 ng of each TDI was spiked onto the filters. No TDI was detected on the backup filter confirming the absence of breakthrough losses.

Next, the recoveries of the TDIs were evaluated as a function of mass loading. Impregnated filters were spiked with 10, 51, 102, 153, and 204 ng of both TDI isomers and then 10 L of room air was drawn through each of the filters. At least four filters were spiked at each mass level. The average recovered masses plotted as a function of the spiked masses are shown in Figures 2 and 3 for 2,4-TDI and 2,6-TDI,

respectively. The relationships had small negative intercepts and linear regression coefficients of at least 0.998, indicating that the recoveries were independent of the spiked masses. The slopes of the regression lines and 95% CIs were 0.775 ± 0.064 for 2,4-TDI and 0.693 ± 0.041 for 2,6-TDI. These slopes are the best indicators of the recoveries from vapor-spiked filters. Therefore, the overall recovery of 2,4-TDI from vapor-spiked filters is 77 ± 6 percent. Because of the higher uncertainty in the diffusion rate for 2,6-TDI, the overall recovery for this compound is 69 ± 10 .

The 95% CI for the measured masses of replicate filters was less than ± 10 percent of the average value at each mass loading with the exception of the 10-ng spike level. The higher uncertainty at this level may have been attributable to the difficulty of accurately controlling sampling at the very short time interval needed to achieve the low loading.

To evaluate the cause of the low recoveries, we set up a second vapor generation system using a commercial permeation source (Part No. 107-030-7480, VICI Metronics) containing an 80/20 mixture of the 2,4-TDI and 2,6-TDI isomers. The certified permeation rate for the combined isomers was 13.6 ± 2.0 ng min⁻¹ at 50° C. Five days after installing the source, the measured emission rate was 24.6 ng min⁻¹. It was assumed that equilibrium had not been reached. Measurements were again made with replicate impregnated filters after the source had been in continuous operation for 57 and 77 days. No corrections were made for possible incomplete recoveries. The measured rates were 14.6 ± 2.1 and 16.2 ± 0.8 ng min⁻¹, respectively, which are within or narrowly out of the certified range. These results suggest that the apparent low recoveries discussed above may actually have been due to problems with the diffusion sources rather than to recovery or sample preparation losses.

The storage stability of vapor-spiked filters was evaluated at spike levels of 51 and 153 ng of each TDI. Eight spiked filters at each mass loading were stored at room temperature (22 - 25° C) and eight were stored in the freezer at -15° C. Storage times were one and two weeks. Each filter was placed in a 20-mL screw-top vial wrapped with aluminum foil to prevent exposure to light. The room-temperature stored filters produced consistent results for both isomers. The average recoveries and 95% CIs for 2,4-TDI were 69 ± 6 percent and 74 ± 3 percent for the first and second weeks, respectively. The corresponding recoveries for 2,6-TDI were 65 ± 4 percent and 70 ± 3 percent. Freezer storage introduced higher uncertainties possibly due to condensation of water vapor. The average recoveries and 95% CIs were 71 ± 8 percent for 2,4-TDI and 69 ± 8 percent for 2,6-TDI after freezer storage for one week. After two weeks storage, the uncertainties were even higher (about ± 20 percent). The performance of the method is summarized in Table I.

Emissions of TDIs from Polyurethane Foam

Exposures to TDIs from the handling or use of flexible polyurethane foam products can result if there are emissions of unreacted monomers or if there is thermal degradation as might occur during cutting processes. Any such exposures would be expected to be relatively low but may be significant for sensitive individuals under some circumstances.

A sample of prime polyurethane carpet cushion was collected from the production line at a manufacturing facility. It was sealed in a Tedlar bag and shipped to the laboratory for analysis. A piece of the cushion sample (0.2 x 0.3 m) was placed in a silanized 3.4-L glass chamber at 23° C and ventilated with dry nitrogen at 0.3 L min⁻¹. A 54-L air sample was collected on an impregnated filter at the exit of the chamber over an elapsed time interval of 3 - 6 hours. The cushion sample did not emit TDIs at detectable levels ($<0.2 \mu\text{g m}^{-2} \text{h}^{-1}$) under these conditions. The measurement was repeated with the chamber temperature held at 100° C. This elevated temperature is a favorable condition for releasing unreacted TDI monomers from polyurethane foams (6). An integrated air sample was collected over 12 hours starting at the time the chamber was placed in the oven. 90 ng of 2,6-TDI was collected over this period. This is equivalent to a level of unreacted 2,6-TDI in the foam of $1.4 \mu\text{g m}^{-2}$ (2.6 ppb by weight). The emission of 2,6-TDI and not 2,4-TDI is consistent with measurements made in foam manufacturing facilities which show that 2,6-TDI is often the predominant isomer in finishing areas (2,7). This is attributed to the greater reactivity of 2,4-TDI.

CONCLUSIONS

The described analytical method provides an improved ability to measure low-level concentrations of TDIs in air. The very low limit of quantitation of 0.02 ppb for a 15-L sample is largely attributable to the combined use of a C₁₈ base-deactivated silica column and 25-mm filters impregnated with only 50 µg of 1-2PP. As a result, the TDIUs were chromatographically well separated from the initial peak of unreacted 1-2PP and from each other. Tedious sample preparation procedures to selectively remove excess 1-2PP or to concentrate sample extracts were thus avoided. The use of a filter holder with a silanized glass inlet and the use of disposable labware significantly reduced sample losses and cross contamination problems. The results suggested that there was a problem with the generation of known low concentrations of TDI vapors. If this problem can be satisfactorily resolved, it may be possible to demonstrate that the method has near quantitative recoveries.

The improved method has general applicability for industrial hygiene surveys in manufacturing facilities. It is particularly well suited for measurements in locations where concentrations may be below occupational guidelines. Such locations include sites where finished flexible polyurethane foam is handled, as well as offices, schools and residences where low levels of TDIs may be present as the result of emissions from polyurethane foams or other polyurethane products. The method also shows promise as a screening procedure for quantifying residual TDI monomers emitted from finished products.

ACKNOWLEDGMENTS

The authors gratefully acknowledge the assistance of Matthew L. Albert in the development of the method. The project was funded with Federal funds from the U.S. Consumer Product Safety Commission and the Office of Atmospheric and Indoor Air Programs of the U.S. Environmental Protection Agency under interagency agreements, CPSC-IAG-92-1134 and CPSC-IAG-92-1120, as well as the Assistant Secretary for Conservation and Renewable Energy, Office of Building Technologies, Building Systems and Materials Division of the U.S. Department of Energy under Contract DE-AC03-76SF00098. The content of this publication does not necessarily reflect the views of these agencies, nor does mention of trade names, commercial products, or organizations imply endorsement or sanction by these agencies.

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Table I. Performance of Improved Method for TDIs

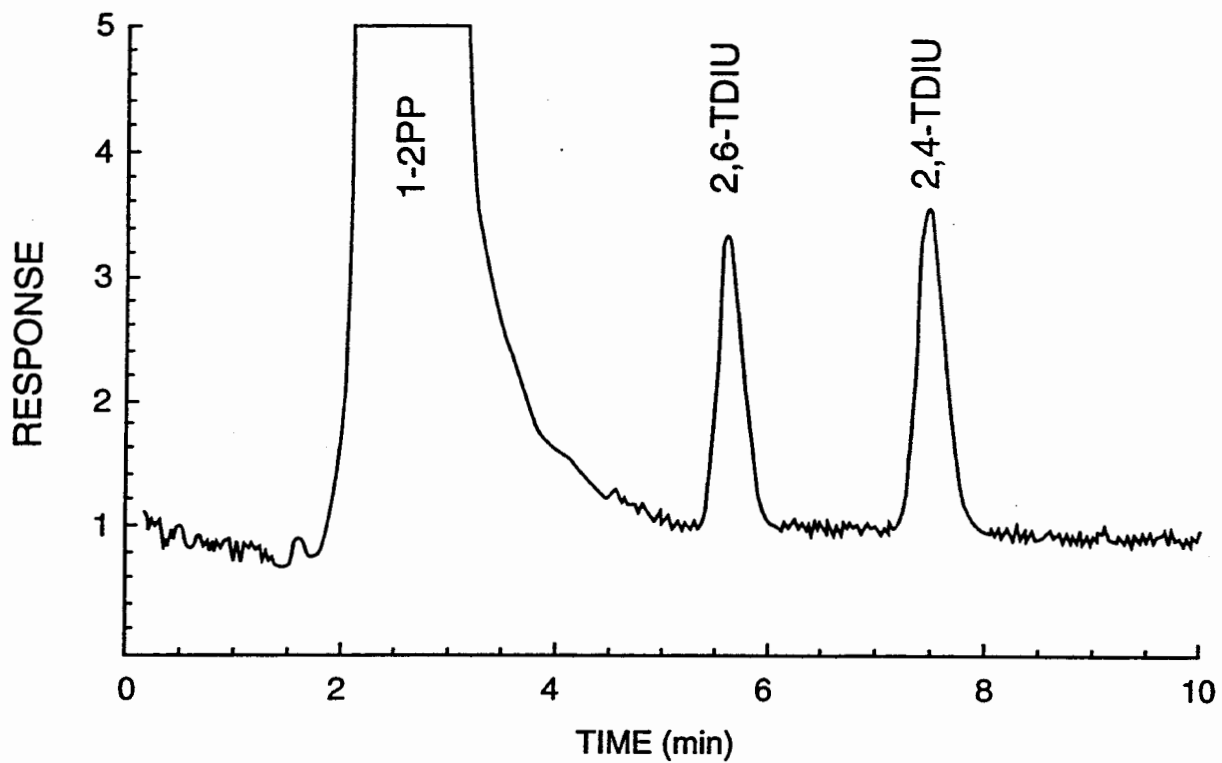
<i>Parameter</i>	<i>Value</i>
Filter capacity	27,000 ng TDI
Lower limit of quantitation	2 ng TDI per sample
Lower limit of quantitation for 15-L sample	0.13 $\mu\text{g m}^{-3}$ or 0.02 ppb
Linear calibration range	7 - 600 pg TDI per injection
Breakthrough	None
Recovery efficiencies (\pm 95% CI)	77 \pm 6% for 2,4-TDI 69 \pm 10% for 2,6-TDI
Precision (95% CI)	\sim 10%
Storage stability	2+ weeks at room temperature

Figure Captions:

Figure 1. HPLC analysis of extract of impregnated filter vapor spiked with 51 ng each of 2,4-TDI and 2,6-TDI.

Figure 2. Linear regression and 95% confidence intervals for average recovered masses of 2,4-TDI plotted as a function of masses vapor spiked onto impregnated filters.

Figure 3. Linear regression and 95% confidence intervals for average recovered masses of 2,6-TDI plotted as a function of masses vapor spiked onto impregnated filters.



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Figure 1. HPLC analysis of extract of impregnated filter vapor spiked with 51 ng each of 2,4-TDI and 2,6-TDI.

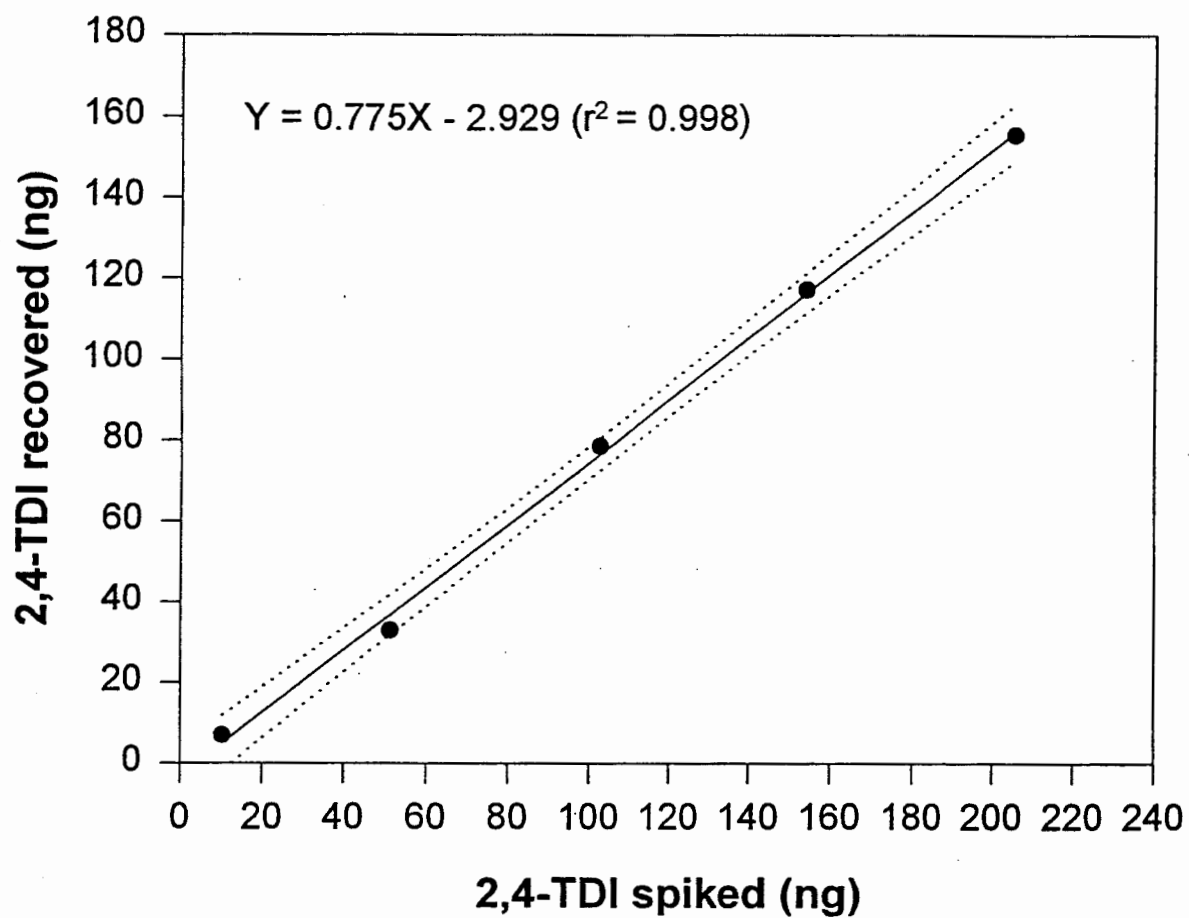


Figure 2. Linear regression and 95% confidence intervals for average recovered masses of 2,4-TDI plotted as a function of masses vapor spiked onto impregnated filters.

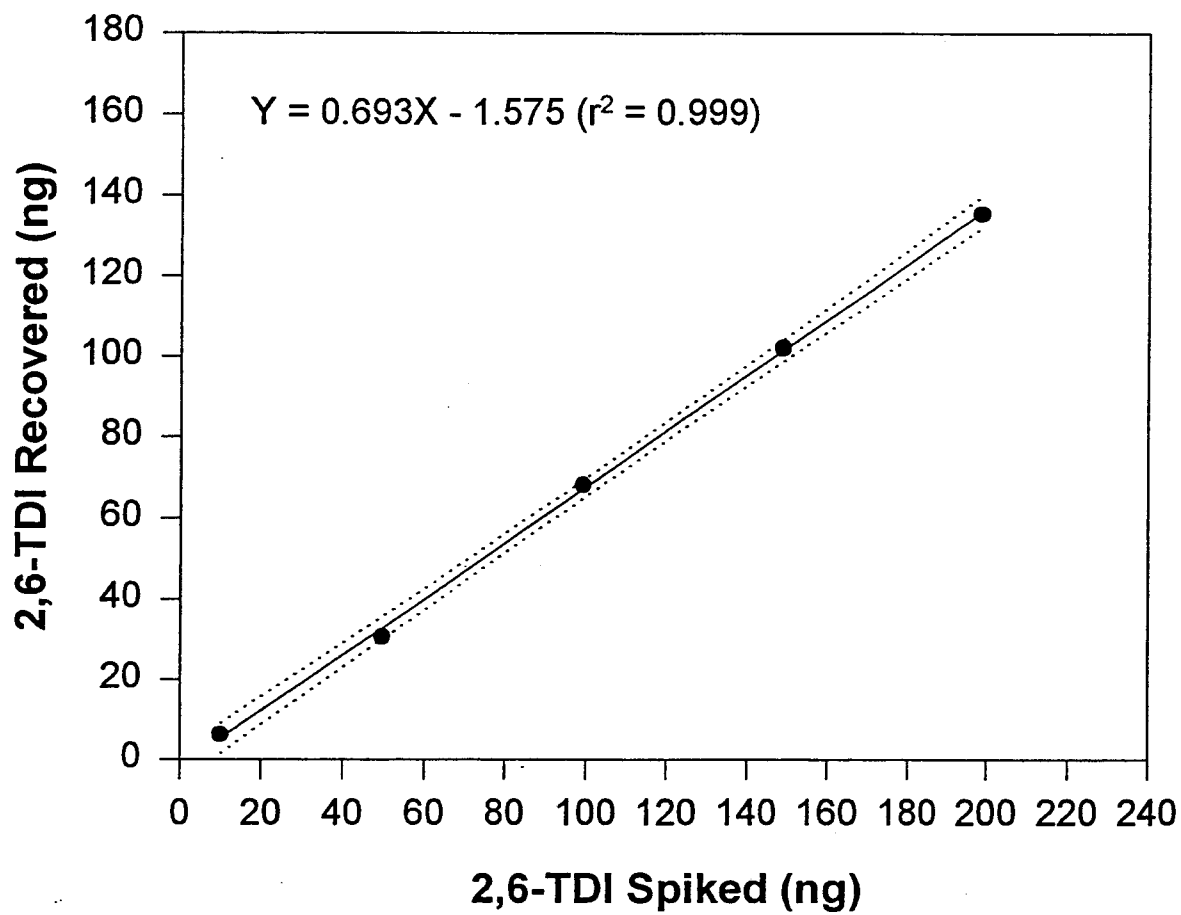


Figure 3. Linear regression and 95% confidence intervals for average recovered masses of 2,6-TDI plotted as a function of masses vapor spiked onto impregnated filters.

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